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SPECIFICATION FOR FENITROTHION, TECHNICAL

(Second Reprint FEBRUARY 2001)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 4 *December* 1969

Indian Standard

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AMENDMENT NO. 1 APRIL 1989
TO
IS : 5280 - 1969 SPECIFICATION FOR FENITROTHION, TECHNICAL

(*Page* 4, *clause* **2.2**, *Table* 1, *Sl No.* (v), *col* 2 *and* 3) — 'Substitute '27°/27°C' *for* '25°C' and '1·310 — 1·320' *for* '1·32'.

(*Page* 9 *and* 11) — Substitute '27°C' *for* '25°C' wherever it occurs.

(AFCDC 6)

Printed at New India Printing Press Khurja India

Indian Standard

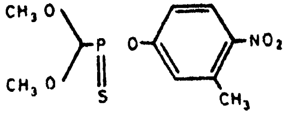
SPECIFICATION FOR FENITROTHION, TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 26 September 1969, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Fenitrothion, technical, is employed in the preparation of insecticidal formulations used for the control of pests of agricultural and public health importance.

0.2.1 Fenitrothion is the common name accepted by the International Organization for Standardization for the pesticide chemical containing *OO*-dimethyl *O*-(3-methyl-4-nitrophenyl) phosphorothioate (*see* Note) as its active ingredient. The empirical and structural formulæ and the molecular weight of the compound are as indicated below:

Empirical Formula	*Structural Formula*	*Molecular Weight*
$C_9H_{12}O_5PSN$		277·24

NOTE — The other chemical name accepted by the International Organization for Standardization for this pesticide is dimethyl 3-methyl-4-nitrophenyl phosphorothionate.

0.3 Taking into consideration the views of producers, consumers, testing authorities and technologists, the Sectional Committee responsible for the preparation of this standard felt that it should be related to the manufacturing and trade practices followed in the country in this field.

0.4 This standard is one of a series of Indian Standards on pesticides and their formulations.

0.5 This standard contains clauses **E-2.3** and **E-3.4** which call for an agreement between the purchaser and the vendor.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of test for fenitrothion, technical, employed in the preparation of insecticidal formulations.

2. REQUIREMENTS

2.1 Description — The material shall be amber coloured, slightly oily liquid, free from extraneous impurities or modifying agents and it shall be insoluble in water.

2.2 The material shall comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR FENITROTHION, TECHNICAL

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Appendix	Indian Standard
(1)	(2)	(3)	(4)	(5)
i)	Active ingredient (*see* 0.2.1), percent by weight, *Min*	95	A	—
ii)	Material insoluble in acetone, percent by weight, *Max*	0·5	B	—
iii)	Moisture content, percent by weight, *Max*	0·5	—	IS : 2362-1963*
iv)	Acidity as (H_2SO_4), percent by weight, *Max*	0·5	C	—
v)	Specific gravity at 25°C	1·32	D	—

*Method for the determination of water by the Karl Fischer method.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in mild steel containers lined with phthallic, phenolic or epoxy resins. For retail packs, galvanized tin containers lined with phthallic, phenolic or epoxy resins shall be used.

---

*Rules for rounding off numerical values (*revised*).

3.2 Marking — The containers shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;
b) Name of the manufacturer;
c) Date of manufacture;
d) Batch number;
e) Net weight of contents;
f) Active ingredient content; and
g) The minimum cautionary notice worded as under:

'HIGHLY POISONOUS. HANDLE WITH CARE. KEEP AWAY FROM FOODSTUFFS AND ANIMAL FEEDS. DO NOT TOUCH OR INHALE THE CONTENTS. WHILE HANDLING THE CONTENTS, USE RUBBER GLOVES AND FACE MASK. USE OF EMPTY CONTAINERS DANGEROUS. IN CASE OF POISONING, CALL A PHYSICIAN. ATROPINE AND OXYGEN ARE USEFUL IN TREATMENT. AVOID SKIN CONTACT.'

3.2.1 The product may also be marked with Standard mark.

3.2.1.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Appendix E.

5. TESTS

5.1 Tests shall be carried out as prescribed in the appropriate appendices given in col 4 and 5 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960[*]) shall be employed in tests.

NOTE — 'Pure Chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

---

[*] Specification for water, distilled quality (*revised*).

APPENDIX A

[*Table* 1, *Item* (i)]

DETERMINATION OF *OO*-DIMETHYL *O*-(3-METHYL-4-NITROPHENYL) PHOSPHOROTHIOATE

A-1. PRINCIPLE OF METHOD

A-1.1 The sample is dissolved in ether and the most likely impurity, 4-nitro-*m*-cresol, is extracted with mild alkali. The ether layer is treated with zinc and an acetic acid - hydrochloric acid mixture, and the amino groups are titrated with standard sodium nitrite solution.

A-2. REAGENTS

A-2.1 Acetic Acid - Hydrochloric Acid Mixture — Mix 9 volumes of glacial acetic acid with one volume of concentrated hydrochloric acid.

A-2.2 Standard Sodium Nitrite Solution — 0·1 M, standardized against sulphanilic acid. Dissolve 6·90 g of sodium nitrite in distilled water and make up to one litre with distilled water. Standardize as follows:

> Weigh accurately 0·40-0·45 g of anhydrous sulphanilic acid (analytical grade) into a 400-ml tall-form beaker. Add 80 ml of distilled water, 10 ml of concentrated hydrochloric acid, 30 ml of glacial acetic acid and 5 g of sodium (or potassium) bromide. Cool the mixture to 0°-10°C by the addition of clean, shaved ice and stir mechanically. Titrate at 0°-10°C with the 0·1 M sodium nitrite as rapidly as the spot test permits. Near the end-point, add the sodium nitrite in four-drop portions.
>
> $$\text{Normality of 0·1 M sodium nitrite} = \frac{a \times 5·774}{b}$$
>
> where
>
> a = weight (in g) of sulphanilic acid used, and
>
> b = volume (in ml) of 0·1 M sodium nitrite required.

A-3. PROCEDURE

A-3.1 Extraction of Free 4-Nitro-*m*-Cresol — Weigh accurately about 1 g of the sample and transfer the same in a 200-ml separating funnel containing 100 ml of ether. Extract the ether solution four times with 20 ml portions of chilled 1 percent (*w*/*v*) sodium carbonate solution and discard the aqueous layers. The extraction should be done as quickly as possible to prevent decomposition of the active ingredient.

A-3.2 Determination of *OO*-Dimethyl *O*-(3-Methyl-4-Nitrophenyl) Phosphorothioate in Ether Layer — Transfer the ether layer quantitatively to a 400-ml beaker, using small portions of ether. Add 100 ml of the acetic acid - hydrochloric acid mixture and 10 g of iron-free zinc dust. Cover the beaker with a watch-glass and heat gently on a steam-bath until most of the ether has evaporated and the solution is colourless. Add 25 ml of concentrated hydrochloric acid and 75 ml of distilled water to complete the solution of the zinc dust. Cool, wash down the beaker and watch-glass with distilled water, and add five grams of sodium (or potassium) bromide. Cool the mixture to 0°-10°C by the addition of clean, shaved ice and stir mechanically. Titrate at 0°-10°C with the standardized 0·1 M sodium nitrite as rapidly as the spot test permits. Near the end-point, add the sodium nitrite in four-drop portions.

A-3.3 Spot Test — Dip a glass rod into the solution to be tested and then touch the rod quickly to a piece of potassium iodide/starch paper. The end-point is reached when an intense blue-black colour appears immediately and may be obtained repeatedly during a one-minute period without further addition of sodium nitrite.

A-4. CALCULATION

A-4.1 *OO*-dimethyl *O*-(3-methyl-4-nitrophenyl) phosphorothioate content, percent by weight $= \frac{27 \cdot 72\, W N f}{w}$

where

W = volume (in ml) of 0·1 M sodium nitrite required;

N = normality of 0·1 M sodium nitrite;

f = correction factor $\frac{T}{A}$; and

w = weight (in g) of sample;

where

T = quantity (in ml) of 0·1 M sodium nitrite calculated for the nitro-group determination of one gram of a mono-nitro reference material, and

A = quantity (in ml) of 0·1 M sodium nitrite used in the nitro-group determination of one gram of a recrystallized sample of the same substance, following the method described above and using the same reagents.

The correction factor f is intended to allow for errors due to impurities in the reagents, as well as those inherent in the method itself or in its application by a given laboratory. Its value shall lie within the range 0·98 to 1·02.

APPENDIX B

[*Table* 1, *Item* (ii)]

DETERMINATION OF MATERIAL INSOLUBLE IN ACETONE

B-1. REAGENT

B-1.1 Acetone

B-2. PROCEDURE

B-2.1 Weigh 10 g of the material into a clean and dry 250-ml Erlenmeyer flask fitted with a ground-glass joint. Add 150 ml of dry acetone and warm under reflux until all soluble material is dissolved. Filter the solution using a tared Gooch or sintered glass crucible of porosity No. 3, and wash well with more solvent. Dry at 110°C for 30 minutes, cool, and weigh.

B-3. CALCULATION

B-3.1 Matter insoluble in acetone, percent by weight $= \frac{100\,w}{W}$

where

w = weight in g of the residue obtained after drying, and
W = weight in g of the material taken for the test.

APPENDIX C

[*Table* 1, *Item* (iv)]

DETERMINATION OF ACIDITY

C-1. REAGENTS

C-1.1 Acetone

C-1.2 Methyl Red Indicator Solution — aqueous, one percent (w/v).

C-1.3 Standard Sodium Hydroxide Solution — 0·02 N.

C-1.4 Standard Hydrochloric Acid — 0·02 N.

C-2. PROCEDURE

C-2.1 Weigh accurately about 10 g of the material and dissolve it in 25 ml of acetone. Solution may be effected by gently warming if necessary. Add 75 ml of water and titrate immediately with the standard sodium hydroxide solution using methyl red as the indicator.

C-2.2 Carry out a blank determination using 25 ml of acetone and 75 ml of water.

C-3. CALCULATION

C-3.1 Acidity (as H_2SO_4), percent by weight $= \dfrac{4{\cdot}9\ (V-v)\ N}{W}$

where

V = volume in ml of the standard sodium hydroxide solution required for the test with the material,

v = volume in ml of the standard sodium hydroxide solution required for the blank determination,

N = normality of the standard sodium hydroxide solution, and

W = weight in g of the material taken for the test.

C-3.1.1 In case the blank determination shows an alkaline reaction, neutralize it with the standard hydrochloric acid and calculate the acidity as follows:

$$\text{Acidity (as } H_2SO_4\text{), percent by weight} = \frac{4{\cdot}9\ (VN_1 + vN_2)}{W}$$

where

V = volume in ml of the standard sodium hydroxide solution required for the test with the material,

N_1 = normality of the standard sodium hydroxide solution,

v = volume in ml of the standard hydrochloric acid required for the blank determination,

N_2 = normality of the standard hydrochloric acid, and

W = weight in g of the material taken for the test.

APPENDIX D

[*Table* 1, *Item* (v)]

DETERMINATION OF SPECIFIC GRAVITY

D-1. TERMINOLOGY

D-1.1 For the purpose of this standard, the specific gravity of the material shall be the ratio of the weight in air of a given volume of the material at 25°C to that of an equal volume of water at the same temperature.

D-2. METHOD

D-2.1 The specific gravity shall be determined with a specific gravity bottle — Castell Evans modification with ground-in stopper and ground-on cap (*see* Fig. 1) or with a pyknometer tube of the Perkin's type (*see* Fig. 2).

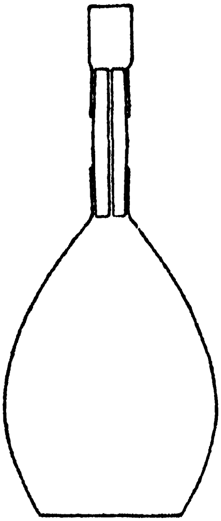


FIG. 1 SPECIFIC GRAVITY BOTTLE (CASTELL EVANS MODIFICATION WITH GROUND-IN STOPPER AND GROUND-ON CAP)

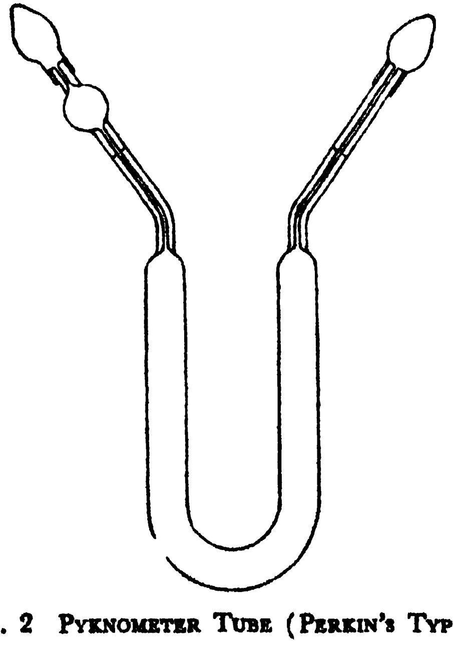


FIG. 2 PYKNOMETER TUBE (PERKIN'S TYPE)

D-3. PROCEDURE

D-3.1 Weigh the clean and dry specific gravity bottle or pyknometer of 25-ml capacity. Fill the tared specific gravity bottle or pyknometer with freshly boiled water and place it in a bath maintained at 25° ± 1°C and allow sufficient time to attain the temperature of the bath. Then insert the ground-in stopper which has also been brought to 25° ± 1°C. Wipe the excess liquid from the stopper and place the cap in position on it. Remove the specific gravity bottle or the pyknometer from the bath, bring to room temperature and weigh. Empty the specific gravity bottle or the pyknometer, clean and dry it, and repeat the operation with the material at 25° ± 1°C.

D-4. CALCULATION AND REPORT

D-4.1 Specify the temperature of testing in the report.

D-4.2 The specific gravity is calculated by the following formula:

$$\text{Specific gravity at } 25°/25°\text{C} = \frac{C - A}{B - A}$$

where

C = weight in g of the specific gravity bottle or pyknometer filled with the material at 25°C,

A = weight in g of the dry specific gravity bottle or pyknometer, and

B = weight in g of the specific gravity bottle or pyknometer filled with water at 25°C.

APPENDIX E

(*Clause* 4.1)

SAMPLING OF FENITROTHION, TECHNICAL

E-1. GENERAL PRECAUTIONS

E-1.0 In drawing, preparing, storing, and handling test samples, the following precautions and directions shall be observed.

E-1.1 Samples shall not be taken in an exposed place.

E-1.2 The sampling instrument shall be clean and dry when used.

E-1.3 Proper precautions shall be taken while drawing samples since the material is poisonous.

E-1.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the receptacles for samples from adventitious contamination.

E-1.5 The samples shall be placed in suitable, clean, dry and air-tight sample receptacles.

E-1.6 The sample receptacles shall be of such a size that they are almost, but not completely, filled by the sample.

E-1.7 Each sample receptacle shall be sealed air-tight after filling and marked with full details of sampling, the date of manufacture, name of the manufacturer and other particulars of the consignment.

E-1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

E-2. SCALE OF SAMPLING

E-2.1 Lot — All the containers in a single consignment of the material drawn from the same batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

E-2.1.1 Samples shall be tested for each lot for ascertaining the conformity of the material to the requirements of this specification.

E-2.2 The number of containers to be selected from the lot shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

(*Clauses* E-2.2. E-2.3, E-3.1 *and* E-3.3)

Lot Size N (1)	No. of Containers to be Selected n (2)
3 to 15	3
16 „ 40	4
41 „ 65	5
66 „ 110	7
Over 110	10

E-2.3 These containers shall be selected at random from the lot. To ensure the randomness of selection, a random number table as agreed to between the purchaser and the supplier, shall be used. In case such a table is not available, the following procedure shall be adopted:

> Starting from any container in the lot, count them as 1,2,3, up to r and so on, in one order, where r is equal to the integral part of the value of N/n; N being the total number of containers in the lot and n the number of containers to be selected (*see* Table 2). Every rth container thus counted shall be separated until the requisite number of containers is obtained from the lot to give samples for test.

E-3. TEST SAMPLES AND REFEREE SAMPLES

E-3.1 Before drawing the test sample, mix thoroughly the contents of each container selected, by shaking or by any other suitable means so as to bring all portions into uniform distribution. Draw small portions of the material from different parts of each container selected (*see* Table 2). The total quantity of the material drawn from each container shall be not less than 300 g.

E-3.2 Mix thoroughly all the material drawn from the same container. A small but equal quantity shall be taken from each selected container and shall be well mixed together so as to form a sample of not less than 250 g. This sample shall be divided into three equal composite test samples, one for the purchaser, another for the supplier, and the third for the referee.

E-3.3 The remaining material drawn from a container (after the quantity needed for the formation of the composite test samples has been taken out) shall be divided into three equal parts. These parts shall be immediately transferred separately to thoroughly dried sample receptacles which are then sealed air-tight, and labelled with all the particulars of sampling given under **E-1.7**. The material in each such sealed sample receptacle shall constitute an individual test sample. These individual test samples shall be separated into three identical sets of individual test samples in such a way that each set has a sample representing each container selected (*see* Table 2). One of these three sets shall be marked for the purchaser, another for the supplier and the third for the referee.

E-3.4 Referee Samples — Referee samples shall consist of one of the composite test samples (*see* **E-3.2**) and a set of individual test samples (*see* **E-3.3**) for this purpose, and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the two.

E-4. TESTING OF SAMPLES

E-4.0 The number of tests mentioned in **E-4.1** and **E-4.2** shall be first conducted on the test samples marked for the purchaser. In contingencies, such as loss, spoilage, or spilling, of the purchaser's test samples or if the supplier so desires, the test samples marked for the supplier may be tested. In case of a dispute, the referee test samples shall be tested and the test results obtained on the referee test samples shall be considered as final.

E-4.1 Tests for description (*see* **2.1**) and determination of active ingredient content shall be conducted on each of the individual test samples.

E-4.2 Tests for the determination of the remaining characteristics, namely, water content, acidity, material insoluble in acetone, and specific gravity shall be conducted on the composite test sample as prepared under **E-3.2.**

E-5. CRITERIA FOR CONFORMITY

E-5.1 A lot shall be declared as conforming to the specification, when:

a) each of the individual test samples satisfies the requirement specified in **2.1**;

b) Each of the test results for active ingredient content satisfies the requirements specified in Table 1. If, however, one or more of the test results does not satisfy the specified requirement, the conformity of the lot shall be ascertained in accordance with **E-5.1.1**; and

c) the test results on the composite test sample satisfy the corresponding requirements given in Table 1.

E-5.1.1 In case one or more of the test results does not satisfy the requirement for active ingredient content, the following procedure shall be adopted for conformity of the material in respect of this characteristic. The mean and the range of the corresponding test results shall be calculated as:

$$\text{Mean } (\bar{X}) = \frac{\text{Sum of test results}}{\text{Number of test results}}$$

Range (R) = Difference between the highest and the lowest values obtained for the test results.

The appropriate expression as shown in col 5 of Table 3 shall be calculated. If the value of this expression satisfies the relevant condition as given in col 5 of Table 3, the lot shall be declared to have satisfied the requirement for active ingredient content.

TABLE 3 CRITERION FOR CONFORMITY

CHARACTERISTIC	TEST RESULTS	MEAN	RANGE	CRITERION FOR CONFORMITY
(1)	(2)	(3)	(4)	(5)
Active ingredient content, percent by weight	—	$\bar{X}$	R	$(\bar{X} - 0{\cdot}6\,R) \geqslant 95{\cdot}0$

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002
Telephones: 323 0131, 323 3375, 323 9402
Fax : 91 11 3234062, 91 11 3239399, 91 11 3239382

Telegrams : Manaksanstha
(Common to all Offices)

Central Laboratory:	**Telephone**
Plot No. 20/9, Site IV, Sahibabad Industrial Area, SAHIBABAD 201010	8-77 00 32

Regional Offices:

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002	323 76 17
*Eastern : 1/14 CIT Scheme VII M, V.I.P. Road, Maniktola, CALCUTTA 700054	337 86 62
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NIT Building, Second Floor, Gokulpat Market, NAGPUR 440010	52 51 71
Institution of Engineers (India) Building, 1332 Shivaji Nagar, PUNE 411005	32 36 35

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*Sales Office is at 5 Chowringhee Approach, P. O. Princep Street, CALCUTTA 700072	27 10 85
†Sales Office is at Novelty Chambers, Grant Road, MUMBAI 400007	309 65 28
‡Sales Office is at 'F' Block, Unity Building, Narashimaraja Square, BANGALORE 560002	222 39 71

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